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FESTSCHRIFT IN HONOUR OF PROF T R ANANTHARAMAN ON THE OCCASION OF HIS 80TH BIRTHDAY

Sliding wear properties of high purity copper in cryogenic environment

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Abstract In an effort to understand the influence of cryogenic environment on the friction and wear properties of metallic materials, we performed a series of experiments on high purity work hardened copper (Cu) samples in liquid nitrogen (LN_2) environment against steel (bearing grade; SAE 52100) at varying loads and sliding speeds. The load was varied between 10 and 20 N and sliding speed was varied between 0.89 and 1.34 m/s. In our experiments, a reduction in the steady state coefficient of friction (μ_F) was noted with increasing load (10, 15, 20 N) at the highest sliding speed of 1.34 m/s. High wear rate of the order of 10^{-4} mm^3/Nm was recorded, which was found to be independent of the load/sliding speed. On the basis of the experimental data and the characteristics of the worn surfaces it is confirmed that significant damage accumulation and plowing-induced material removal contribute to the wear losses. It is noteworthy that oxidative wear or mechanically mixed layer (no transfer from steel counter-body) did not occur to any significant extent under the chosen sliding conditions. The characteristics of the wear damage as a result of cryogenic sliding have been discussed with reference to the prevailing stress conditions and contact temperature.

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Introduction

Among various metallic materials, high purity copper is known to have a favorable combination of electrical, thermal, and corrosion resistance properties [1–4]. Copper also has moderate mechanical strength and good fatigue resistance. As a result, copper finds applications in numerous fields such as electronics, chemical, biomedical, structural, and marine engineering. In many engineering applications, tribological properties of copper determine the performance of the component in service conditions. However, only a small number of studies have been conducted to evaluate the friction and wear behavior of high purity copper [1–7].

Nagasawa and Kato [1] performed a wear study of copper alloy wire against iron-base strip applying an electric current and reported that wear rate essentially depends on temperature and contact pressure. While evaluating the friction and wear properties of copper-graphite brushes, Casstevens et al. [2] found improved wear resistance of ceramic particle-reinforced copper in dry sliding conditions. This is believed to be due to the improvement in hardness because of the strengthening effect from ceramic particles [4, 5]. From these reports, it is also clear that copper with medium to coarse grain sizes is not a preferred material choice for tribological applications, because of its low hardness. While copper has Vickers hardness of the order of 0.37 GPa, hardened steel has the hardness of the order of 7 GPa. However, with the development of fine grain materials, there have also been efforts to produce fine grain copper and evaluate its potential for tribological applications [7–9].

Although the friction and wear properties of several metallic alloys in unlubricated conditions are widely investigated, such understanding in cryogenic environment is rather limited [9–17]. Since wear is a system-dependent property, the friction and wear properties depend on both operating (environment, load, sliding speed, etc.) and material (grain size, hardness, etc.) parameters. In our recent research efforts, we attempt to understand how the wear damage takes place in sub-zero temperature, i.e., LN_2, for a variety of materials, including conventional metallic as well as advanced ceramic materials [12–14]. Self-mated stainless steel (AISI 304), self-mated alumina, and other tribo-couples such as Ti/steel in liquid nitrogen environment showed severe wear of the order of 10^{-4}–10^{-5} mm^3/Nm [12–14]. It is also known that the hardness of pure copper increases at sub-zero temperatures and copper is expected to exhibit improved tribological performance at sub-zero temperatures [11]. Our literature survey indicates that relatively small number of studies was conducted to understand the tribological properties of copper at cryogenic environment [9, 11]. Hughes and Hansen [9] successfully produced ultra-fine grain copper by applying a sliding method and such an effect in combination with sub-zero temperature opens up the possibility of producing fine grains with greater hardness, leading to improved wear resistance when compared to its medium to large grain copper counterpart. In particular, high purity copper and a copper–aluminum (10%) alloy were subjected to sliding against 440C steel in cryogenic fuel environments [9]. The wear occurred by the plastic deformation of a recrystallized layer at the worn surface of the copper samples [9, 11]. Largely wear debris were found in the form of a layered structure adhering to the exit region of the wear scar. Iwabuchi and co-workers reported a reduced μ_F ($\sim$0.25) during fretting of self-mated structural steel at 4 K temperature, while such a reduction was not recorded in copper [16, 17].

In the present study, taking into account the low hardness of the copper, cold-rolled high purity copper samples were used for wear studies at liquid nitrogen (LN_2) temperature against steel. The cryogenic wear tests were performed under varying sliding conditions, i.e., speed between 0.89 and 1.34 m/s and load between 10 and 20 N. The primary goal of the investigation was to evaluate the dominant wear mechanisms of high purity copper in liquid nitrogen environment.

Experimental procedure

Material

In the present study, high purity (99.999%) copper samples were used. Disc-shaped copper samples of dimensions 32 mm diameter and 5 mm thickness were prepared from a cold-rolled sheet that was subjected to 70% reduction in thickness. These samples were mechanically polished using emery papers and alumina suspension to achieve a final surface roughness (R_a) of the order of 1 μm. To reveal the microstructure, a chemical etch was used for approximately 20 s in a solution of 45% ammonium hydroxide, 45% water, and 10% hydrogen peroxide (all by volume). A representative microstructure of the investigated copper sample is shown in Fig. 1. At low magnification, the microstructure of the polished surface is characterized by an inhomogeneous feathery appearance, because of the cold-rolled state. The presence of isolated elongated grains in the form of feather-like structure has been observed only at higher magnifications. Spherical steel balls (bearing grade; SAE 52100, hardness: 63–65 Rc) of 10 mm diameter were used as the counter-body material for all the wear tests. Both copper disc samples and steel balls were ultrasonically cleaned in acetone prior to the sliding wear tests.

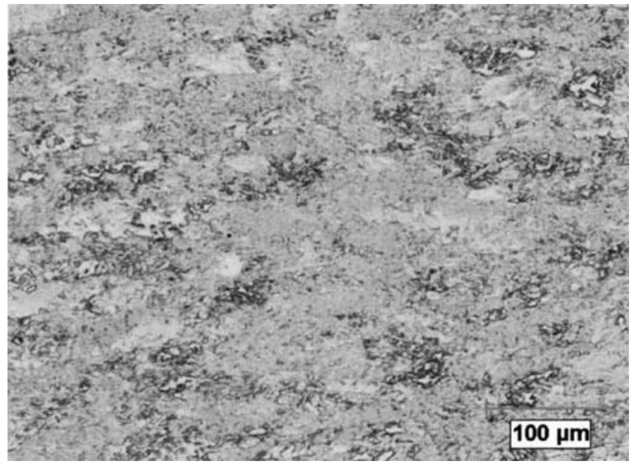

Fig. 1 A representative optical micrograph of the copper samples used in this study. The inhomogeneity seen in the microstructure is a characteristic of the cold-worked material

Wear tests and characterization of worn surfaces

A high speed ball-on-disc tribometer was used in the present study to understand the friction and wear characteristics of high purity copper in LN_2 environment. Figure 2 shows the various components of the tribometer used in this study. In each sliding wear test, the disc sample was in rotational motion, whereas the ball was kept stationary. The normal load was transferred to the ball using a pulley arrangement. The load was applied to the ball which was held stationary in the brass ring. The brass ring was placed in a specially designed ball holder and mated against the rotating disc sample. A custom-made motor was used to achieve a spindle speed as high as 36,000 rpm. Further details of the tribometer can be found elsewhere [18].

Steel balls were kept fixed in the ball holder at three different track radii, i.e., at 10, 12.5, and 15 mm from the

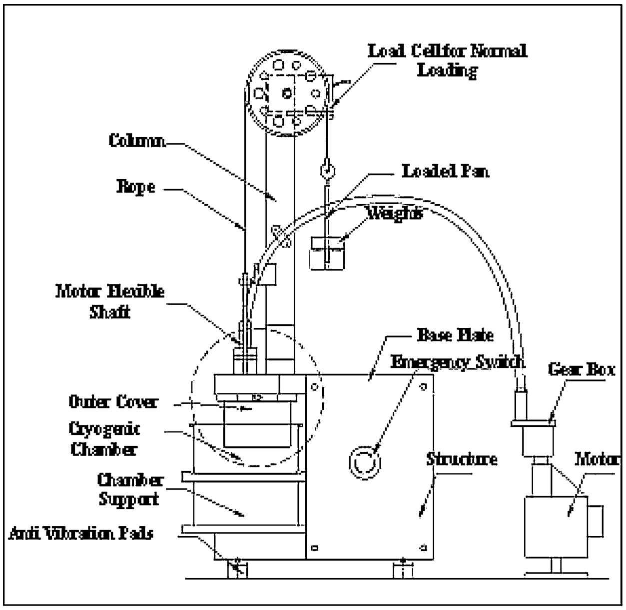

Fig. 2 Schematic diagram showing details of the machine design of cryogenic tribometer. An important part of the machine is ball-on-flat tribological interface, which is located inside the area, marked by a dotted circle

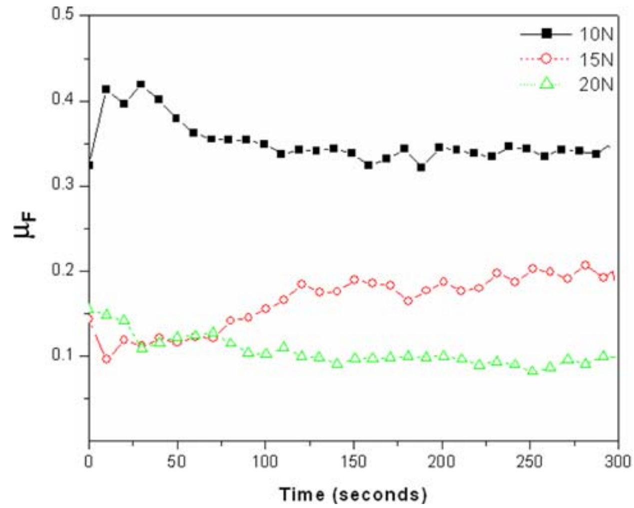

Fig. 3 Representative plots of variation in coefficient of friction (μ_F) during sliding of copper against steel at 1.34 m/s speed in LN_2 environment

central axis and the tests were conducted at a fixed rotational speed of 850 rpm. Such combinations resulted in linear sliding speeds of 0.89, 1.11, and 1.34 m/s, respectively. The ball–disc assembly was immersed in LN_2 bath and the sliding tests were carried out at different loads, i.e., 10, 15, and 20 N. During the test runs, frictional forces were recorded using an electronic sensor to generate online (μ_F) data. The surface profiles of the worn surfaces were acquired using laser surface profilometer (PGK-120, Mahr, Germany) to determine the depth of wear tracks at various test conditions. The wear track width and depth data were further used in calculating wear volume and wear rate. All the experiments were carried out at least three times to check the reproducibility and repeatability of friction and wear data. To understand the wear mechanism of copper, the worn surfaces were characterized using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) (FEI Quanta 200, The Netherlands). On some occasions, the worn surfaces were also analyzed using ZEISS-make SEM-EDS.

Results

Frictional behavior

The frictional behavior of copper/steel tribo-couple in LN_2 environment with varying load and speed conditions was assessed using μ_F data. In all cases, μ_F remained unstable during initial 100 s and thereafter attained nearly steady state until the tests were terminated at 300 s. Figure 3 shows the variation of μ_F as a function of time at three different loads (10, 15, and 20 N) when copper disc was slided against steel ball at a maximum speed of 1.34 m/s. At 10 N load, μ_F increased to a peak value of around 0.42 in the initial 40 s and thereafter gradually decreased to a steady state value of 0.35. In contrast, at 15 N load the μ_F value steadily increased with time and a steady state ($\mu_F \sim 0.2$) was achieved only after 150 s. At the highest load, i.e., at 20 N, the steady state μ_F of 0.1 was achieved rather quickly (within first 25 s) as compared to other cases. It is also clear from Fig. 3 that a small degree of fluctuation in μ_F occurred even in the steady state region for low load conditions and such fluctuations were reduced at the highest load of 20 N.

The average μ_F values in steady state regions are plotted against sliding speed in Fig. 4. The μ_F values of copper/steel couples varied between 0.1 and 0.47 with varying sliding speed and load conditions under LN_2 environment. In all three sliding speeds, a drop in steady state μ_F with higher load is prominent. However, the magnitudes of drops in the steady state μ_F do not show any particular trend with sliding speed. It is possible that a clear trend can be observed if the sliding experiments had been carried out over a wide variation in operating parameters (load, sliding velocity). One can notice that the steady state μ_F corresponding to the 10 N load goes through a peak at intermediate sliding speed, whereas the μ_F corresponding to the 20 N load steadily decreases with increasing sliding speed. These results indicate that at higher load, the μ_F steadily decreases with sliding speed, whereas at relatively low loads this trend is absent.

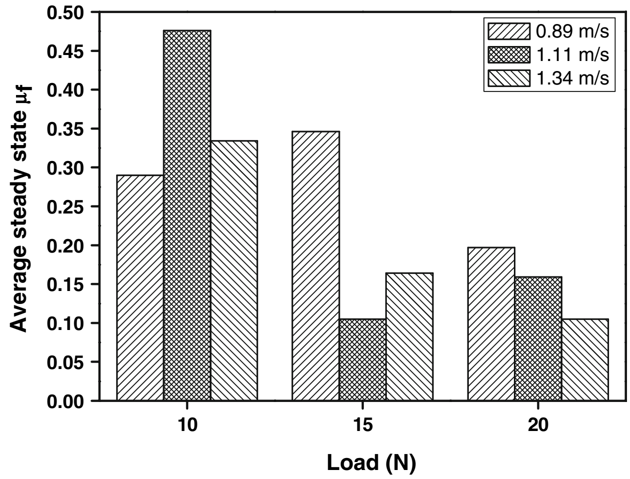

Fig. 4 Average steady state μ_F of copper/steel sliding couple when tested in LN_2 environment

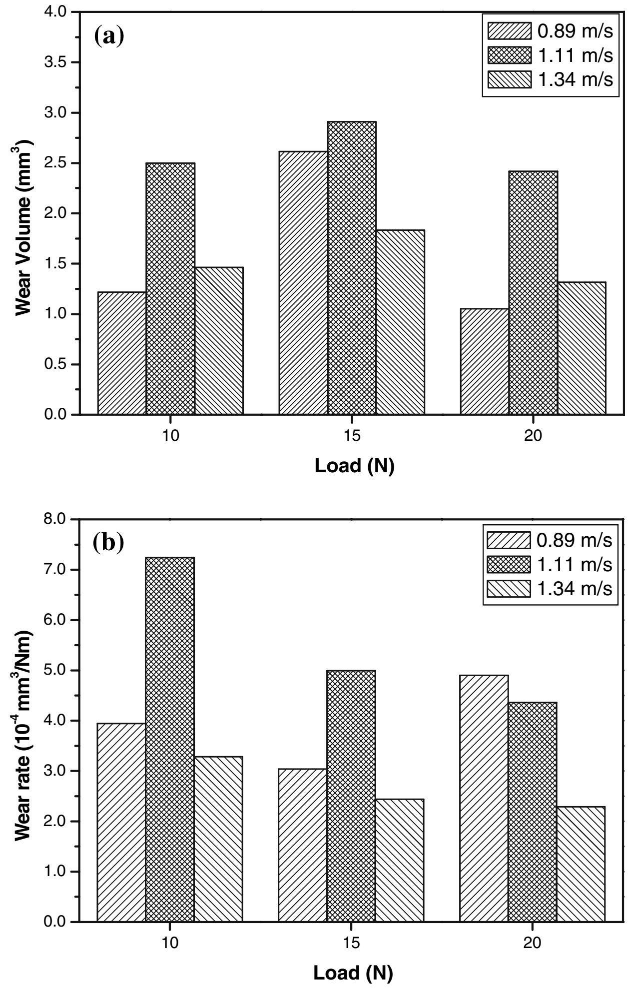

Fig. 5 **a** Wear volume and **b** wear rate of copper slided against steel in LN_2 environment

Wear data

Figure 5 represents the wear volume and the wear rate data of copper as a function of sliding speed at three different loads. For a given sliding speed, wear volume increases with increasing load but this gap reduces with increasing sliding speed (Fig. 5a). There is an overall increase in wear volume at intermediate sliding speed. Similar trend has been noted in case of wear rate (Fig. 5b). However, the wear rate showed a decreasing trend with increasing load at sliding speeds of 1.11 and 1.34 m/s. In this study, the wear rate of copper varied between 0.25 and 0.75 $\times$ 10^{-3} mm^3/Nm, which is one to two orders of magnitude lower than the wear rate reported for copper tested in liquid methane against 440C steel [9]. One would note that the wear volume of copper at an intermediate load is more than in other load conditions.

Figure 6 shows the representative cross sections of the worn tracks as a function of applied load at a sliding speed of 1.34 m/s. The maximum depth of tracks varied from 15 to 30 μm. The depth and the width of the wear tracks increased sharply with the increased load from 10 to 15 N, while such a clear change is absent when load was further increased from 15 to 20 N.

Morphology of the worn surfaces

To understand the dominant mechanisms of material removal, detailed SEM-EDS analysis of worn surfaces of copper disc was carried out and the representative images at different sliding conditions are shown in Figs. 7, 8, 9.

Figure 7 shows SEM micrographs of the worn surfaces of copper samples tested at different sliding speeds at 10 N load. A macroscopic picture of the worn surface of a sample tested at 0.89 m/s sliding speed is shown in Fig. 7a. Figure 7b reveals the rough surface, indicative of the severe damage at the lowest speed of 0.89 m/s. The appearance of sharp grooves on the tribo-layer indicates the occurrence of abrasion. Figure 7c shows the sub-surface damage of the copper surface that has been tested at increased sliding speed of 1.1 m/s. The accumulation of wear debris in the damaged sub-surface is also clear from this photograph. SEM-EDS (inset of Fig. 7c) examination of the surface recorded weak oxygen peak. At the highest sliding speed of 1.34 m/s, the worn surface reveals the traces of abrasion as well as delamination at the sub-surface layers (Fig. 7d). EDS spectra from the tribo-surface again shows weak oxygen peak and strong copper peak (inset of Fig. 7d). No iron peak was detected from steel counter-body at any speed. The sliding tests in ambient conditions were not conducted in the present experimental scheme. However, the occurrence of tribo-oxidation was particularly observed during room temperature sliding tests of several metallic materials [19–21]. Therefore, the minute presence of oxygen along with the absence of iron in the present

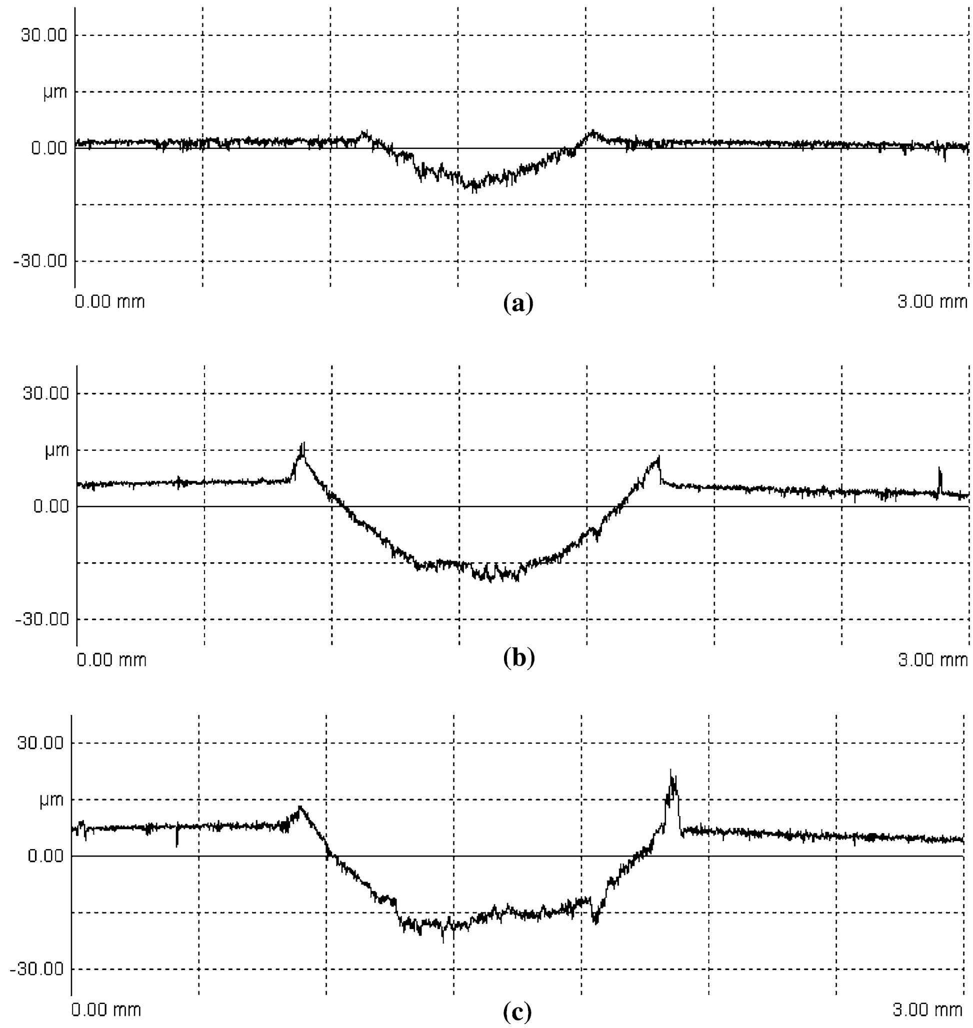

Fig. 6 Representative 2-D surface profiles taken across the wear track on the copper disc after sliding at 1.34 m/s under the load **a** 10 N, **b** 15 N, and **c** 20 N in LN_2 environment

investigation suggest that sliding wear of copper in LN_2 environment occurs primarily due to the mechanical damage.

At 15 N load, surface characteristics of the worn samples are found to be similar to that of 10 N case, in which rough surfaces, because of the severe mechanical damage, are seen (Fig. 8a). High magnification SEM image shows characteristics of abrasion and delamination at the subsurface (Fig. 8b). The delamination of surface layers primarily resulted from cavity formation and such cavities were seen throughout the wear track. Some of the cavities were found to be as large as 125 µm in length. On the other hand, the worn surface of copper sample tested at higher sliding speed shows some discontinuity in the regions that are damaged because of the abrasion and delamination of the layers (Fig. 8c). Large cavities throughout the wear track are visible and the thickness of the damaged layer is of the order of few µm (Fig. 8d).

At 20 N load, delamination of tribo-layer is visible even at the lowest sliding speed of 0.89 m/s (Fig. 9a). Also, the plastic smearing of tribo-layer can be seen in Fig. 9b. However, no evidences of cavitation were noted and the surface was smooth when compared to the surface of the sample tested at 15 N. At increased sliding speed of 1.11 m/s, the worn surface shows wider abrasion grooves and delaminated thick tribo-layers (Fig. 9c). However, the smearing appears to decrease and cavitation starts to occur when compared to that at 0.89 m/s (see Fig. 9b). With further increase in the sliding speed to 1.34 m/s at 20 N load, the surface reveals similar signatures of cavitation and delamination of thicker tribo-layers (Fig. 9d).

Discussion

During dry unlubricated sliding of ductile metals, wear occurs either by mechanical damage of surface and subsurface because of severe localized plastic deformation that may lead to microstructural variation without a considerable changes in the chemistry of the surfaces or by oxidative wear due to the reaction of the mechanically damaged surface/products with the ambient environment [22]. The

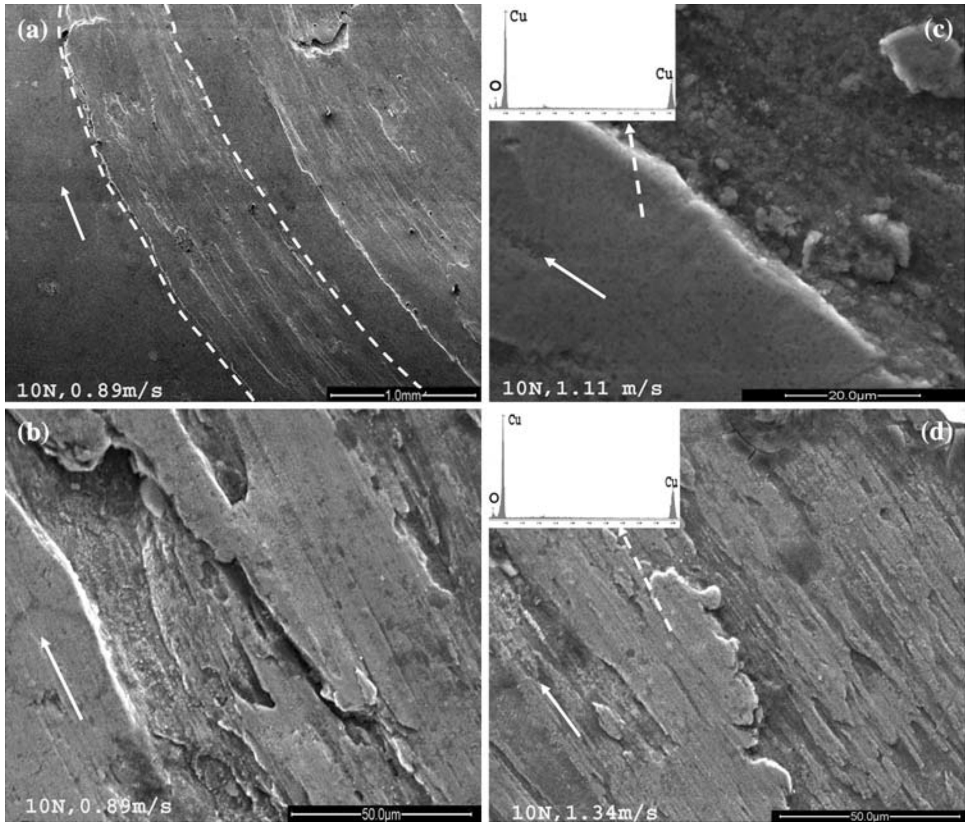

Fig. 7 Worn surface morphology of copper disc after testing at 10 N load with different speeds in LN_2 environment. **a** Macroscopic view of the worn surface, **b** appearance of tribo-layer and abrasion, **c** sub-surface damage and **d** abrasion and delamination. The weak oxygen peaks and no iron peaks recorded in the EDS analysis of surfaces in cases (**c**) and (**d**) indicate the absence of tribo-oxidation and also iron. Arrow indicates sliding direction

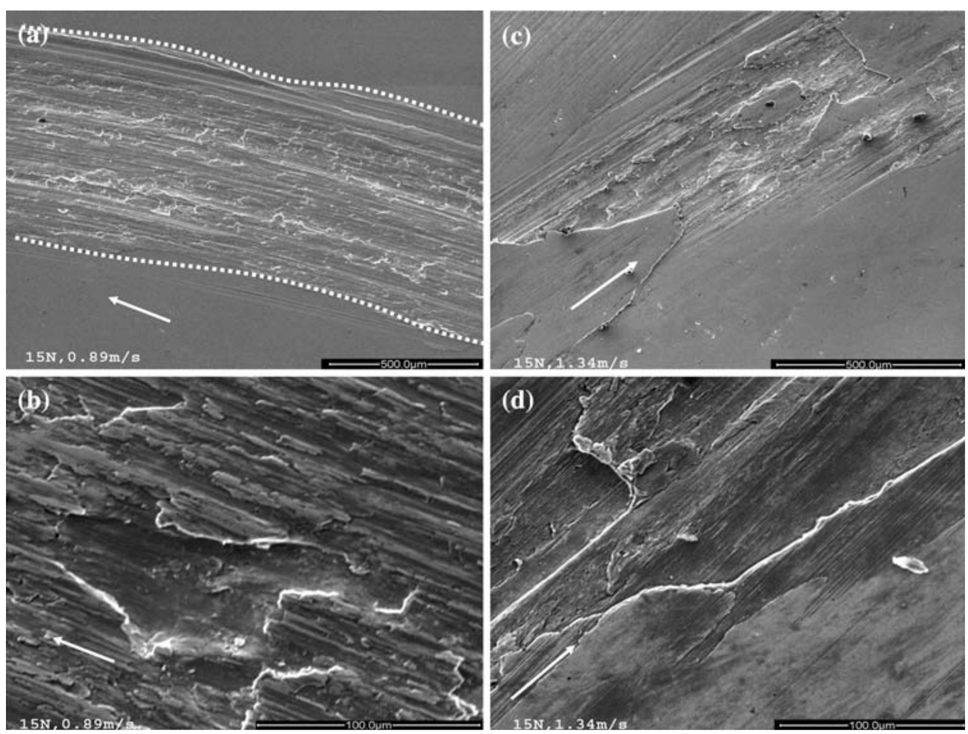

Fig. 8 Secondary electron (SE) images of worn surfaces of copper disc after sliding at 15 N load at different speeds against steel in LN_2 environment. **a** Severe damage with rough surface, **b** cavity-induced delamination, **c** damaged surface and local discontinuity in tribo-layer, and **d** appearance of large cavities throughout the wear track. Arrow indicates sliding direction

latter process is significant in generating third-body material with a difference in chemical composition against base material. Further, physico-chemical behavior and, in particular, the reactivity of such third-body material in the selected sliding conditions are of importance for the subsequent transfer or mixing, thereby affecting further wear [22–24]. However, it has been recognized that the total wear of a system is a result of simultaneous occurrence of the

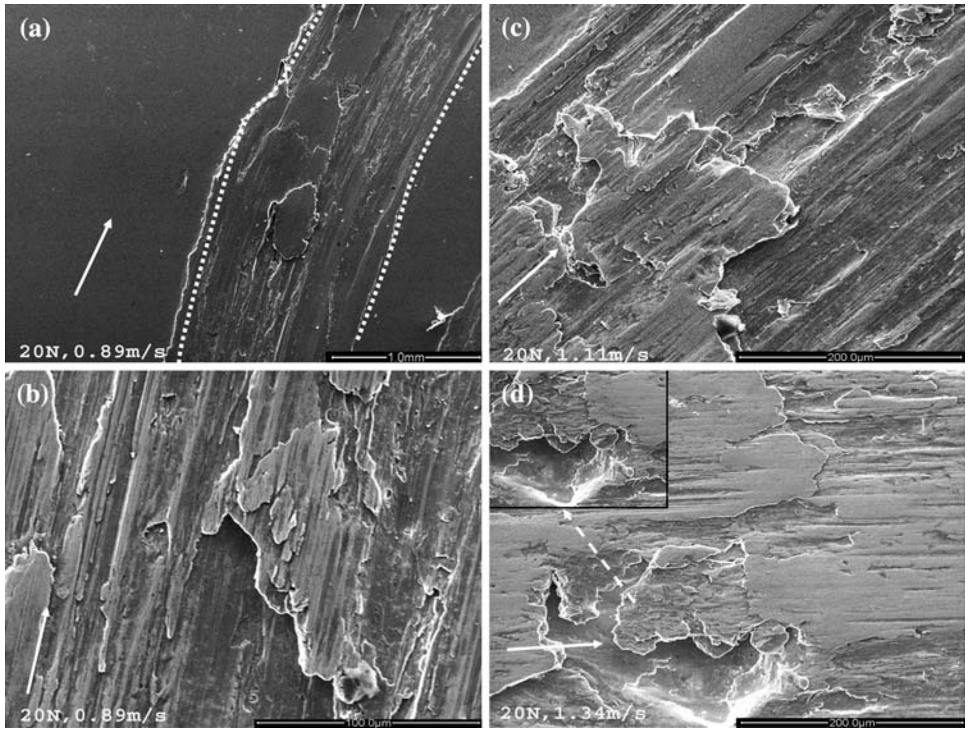

Fig. 9 Worn surface morphology of copper disc that has undergone sliding at 20 N load at different speeds against steel in LN_2 environment. **a** Delamination even at the lowest speed, **b** plastic smearing of tribo-layer, **c** wider grooves, and **d** the cavitation and delamination in tribo-layer. Arrow indicates sliding direction

above processes that makes the entire wear process a rather complex phenomenon. In an earlier work, the wear of ductile copper is recognized to accompany the formation and removal of fine-grained recrystallized layers with the sub-surface being plastically sheared, when tested against steel counter-body in liquid helium temperature [25].

From the present experimental results, it can be summarized that at low load and low speed conditions, copper surface is severely damaged by mechanical means such as abrasion or plowing action of the debris and delamination of the surface layers, while at higher load and high speed conditions, discontinuous delamination with large cavities was found in the entire wear track. EDS analysis of worn surfaces in LN_2 environment did not show any substantial evidences of oxidation or material transfer from the counter-body to the copper sample, indicating occurrence of abrasion and mechanical damage, rather than oxidative wear, as major damage mechanism.

It is also important to correlate the dominant material removal mechanisms to certain phenomenological factors responsible for influencing friction and wear properties in the LN_2 environment. Such correlations can be understood in the light of the material behavior at the tribo-couple interface and, in particular, under the influence of LN_2 environment.

During wear in LN_2 environment, one needs to consider the following basic factors for the interpretation of results. It is believed that the contact surface temperature (reflected in the flash temperature, which is discussed in the next section) can to some extent be reduced because of the rapid heat extraction (heat transfer in LN_2) from the friction surfaces. This is primarily because of high thermal conductivity of copper. Also, the unavailability of oxygen because of the presence of LN_2 blanket is expected to prevent formation of surface oxide layers, as it may otherwise happen during oxidative wear of metallic materials under unlubricated dry sliding conditions. Further, the hardening of the test materials as well as counter-body because of the reduced temperature in the LN_2 bath would also influence the wear behavior.

In the present study of copper slided against steel in LN_2 environment, our aim was to particularly understand the following issues: (a) how does the LN_2 environment influence the friction-induced heat generation at the tribo-interface? (b) whether increase in load or speed results in any transition in the wear mechanisms of copper? and also (c) how does deformation or fracture at sub-zero temperature contribute to the sliding wear properties of copper?

To understand these issues, a rationalized assessment of the experimental results is presented on the basis of two important phenomena, i.e. (i) temperature rise (termed as 'flash temperature' in the following sections) because of the friction at the tribo-contact and the (ii) stress-assisted deformation.

Thermal aspect

An important consideration in the discussion of the wear mechanisms is the estimation of the flash temperature, developed at the tribo-contact in sliding wear test. This is particularly relevant at high sliding speed, e.g., 1 m/s as used in this study. In the present case, flash temperature is calculated using the model proposed by Kong and Ashby [26], because of the difficulty involved in the direct measurement of flash temperature in a dynamic test in cryogenic environment.

From the present experimental conditions of copper sliding against steel in LN_2 environment, the estimation on the basis of Kong-Ashby model indicated that the flash temperatures at the mating interface can vary between −165 and −180 °C. Such low values of flash temperature are in line with high thermal conductivity of copper and steel and also rapid heat transfer from the tribo-contact to the liquid nitrogen. Since thermal conductivity of copper does not vary appreciably between the ambient and the cryogenic test temperatures, the room temperature thermal conductivity value (400 W/mK) was used, while the thermal conductivity of polycrystalline iron (175 W/mK) was used in case of steel counter-body for estimating flash temperature [27]. It is important to note that the heat diffusion distance parameters used in the calculation take the heat transfer from the tribo-contact to the LN_2 bath into account.

The estimated flash temperatures being slightly higher than the boiling point of LN_2, the possibility of thick oxide layer formation during sliding tests in LN_2 environment was low. In fact, SEM-EDS results support this view because only weak oxygen peaks were recorded. To verify this result, similar SEM-EDS studies were carried out on virgin copper samples as well. Presumably, LN_2 environment acts as a blanket and minimizes oxygen flow from the atmosphere to the friction surfaces and prevents the occurrence of tribo-chemical reactions from taking place.

Since the μ_F of the investigated tribo-system is much higher than 0.1 at low load of 10 or 15 N, the possibility of any lubricating effect from LN_2 is remote. However, significantly low value of μ_F of the order of 0.1 at the highest load of 20 N is measured in LN_2 at a sliding speed of 1.34 m/s. The formation of relatively smooth wear track at such severe sliding conditions is possibly the reflection of the favorable frictional property in LN_2. However, the underlying causes of reduced μ_F at higher load and increasing sliding speed are not well understood.

As discussed before, wear tests of copper samples do not seem to form thick oxide layers at any of the sliding conditions, but the development of the plastically deformed tribo-layers is evident from the SEM images of the worn surfaces (Figs. 7, 8, and 9). It is important to note that the copper surfaces are largely able to retain the plastically deformed tribo-layers up to fairly large sliding speeds and the underlying metal is being prevented from further wear, leading to moderate material removal from the worn surface. Fine microcracks and delamination in the form of cavities of tribo-layers at certain locations did not seem to increase the wear rate even at higher sliding speeds.

Tribo-mechanical stress-assisted deformation

In this sub-section, we focus our discussion on the development of plastically deformed tribo-layer and its characteristics in the context of tribo-mechanical stress conditions. Initially, we have assessed whether the operating conditions induce plasticity at the tribo-contact of copper and steel in LN_2 temperature. It is known that the load to initiate yield at the tribo-contact is [28]

$$W_Y = 21.17 R^2 \, Y \, (Y/E^*)^2 \tag{1}$$

where Y is the yield strength of the test piece, E^* is the effective elastic modulus, and R is the radius of the ball counter-body.

The effective elastic modulus E^* can be calculated using the following equation [29]:

$$1/E^* = (1 - \nu_1^2)/E_1 + (1 - \nu_2^2)/E_2 \tag{2}$$

where E_1 and E_2 are the elastic modulus and ν_1 and ν_2 are the Poisson's ratio of the mating solids.

Taking the Poisson's ratios to be equal to 0.3 for both the mating materials and elastic modulus of the steel and copper to be 210 and 130 GPa, respectively, the value of W_Y is estimated to be 2.8 N. Since all the sliding tests in the present study are conducted at a load of 10 N or above, it is clear that the plasticity is introduced to the copper samples at the tribo-contact. For the case of plastic contacts with ductile metals as mating solids, the maximum contact pressure (p_o) can be obtained by applying Von Mises or Tresca's condition [28]. Adopting this, one can arrive at the following relationship (assuming, Poisson's ratio $\nu = 0.3$)

$$(p_o) = 1.6Y \tag{3}$$

where Y is the yield strength in pure tension. Taking the yield strength of cold-worked Cu to be 345 MPa, the maximum contact pressure is estimated to be 552 MPa. It is also known that the maximum shear stress (τ_{max}) for a circular contact is 0.31 p_o for $\nu = 0.3$ [28]. In the present case, the τ_{max} therefore reaches a value of 171.1 MPa.

Under such stress conditions, the tribo-contact point would emit large number of dislocations and consequently generate plastically deformed tribo-layers at the worn surfaces. Therefore, the plastic deformation at wear track and underneath is inevitable. As a result, the observed plowing and smearing of the tribo-layers are conceivably the consequence of such deformation of the tribo-surfaces. The average hardness (Rockwell F, R_F) of the tribo-layers was found to be higher than the hardness of the unworn surface (outside the sliding track). The hardness of the tribo-layers was 96 R_F as against 88 R_F measured on the virgin surface of cold-rolled copper.

In the present case, the flash temperature being fairly low (−165 to −185 °C) and the maximum sliding speed being 1.34 m/s, it is unlikely that dynamic recrystallization would take place in the plastically deformed tribo-layers as seen in the high strain rate cases (6.2–12.4 m/s) in liquid methane environment [25]. However, dynamic recovery can take place even at such low temperatures, because of the rearrangement of dislocations that lowers the strain energy of the deformed structure. This may lead to the formation of cell structure [30]. In fact, Dragomir et al. [31] conducted a systematic X-ray study of dislocation structure in high purity copper subjected to rolling at LN_2 temperature. These samples were cryo-rolled to achieve reduction in thickness between 67 and 97%. The median crystallite size for 67% cryo-rolled copper was found to be approximately 70 nm with relatively large variance. The median crystallite size reduced to approximately 20 nm for 97% cryo-rolled copper with much reduced variance. It is important to remember that the magnitude of the strain in the present case of cryo-sliding wear study of copper was larger than the strain used in the cryo-rolling study [31]. Considering the high purity of copper and the large strain involved in the cryo-wear, the extent of this dynamic recovery process can be significant. Traditionally, the kinetics of dynamic recovery of a metal can be monitored by calorimetric measurements, resistivity measurements, mechanical measurements (hardness or yield stress), and also microstructure evaluation (using transmission electron microcopy) [30]. In this case, the tribo-layer being very thin, any selective measurement of resistivity in the tribo-layers and also preparation of samples for transmission electron microscopy without causing damage to the microstructure were ruled out. However, an alternative method of imaging microstructure in thin surface layers would be discussed in the last section. Therefore, the hardness of the tribo-layers has been measured and a possible role of recovery on the limiting work hardening capability of copper and preventing the hardness increase in the tribo-layers has been discussed.

The ambient temperature hardness of the virgin copper sample was R_F 88, which increased to a value of R_F 98 at LN_2 temperature. After conducting cryo-wear tests at LN_2 temperature and bringing the samples to the ambient temperature, the average hardness of the tribo-layers at RT was recorded to be R_F 96. Even after undergoing severe plastic deformation at LN_2 temperature, the hardness of the tribo-layers increased marginally by 8 units of R_F. One may argue that the increase in the hardness of tribo-layer by 8 units of R_F is because of the plastic deformation without the occurrence of any recovery process. However, considering the severe plastic strain involved in a wear test at LN_2 temperature, one would expect a significant rise in the hardness because of the work hardening of copper, which has not happened in this case. To understand this phenomenon of limited work hardening and marginal increase in the hardness, we wanted to check if this phenomenon is equally true at ambient temperature tests in a simpler mode of deformation. Hence, we conducted a number of ambient rolling experiments on the virgin copper sample that had 70% deformation (reduction in thickness). On further rolling, the average hardness values of 80, 90, and 95% rolled samples were found to be R_F 91.5, 94.5, and 94.0, respectively. Therefore, the average hardness of the virgin copper sample went up by about 6 units of R_F and then saturated with further deformation. These results imply that irrespective of the ambient or LN_2 temperature, the hardness increase (here 6–8 units of R_F) was limited by a restoration process, which took place concurrently with the deformation. In this case, a dynamic recovery process because of the dislocation rearrangement is believed to be the reason for the limiting work hardening capability of high purity copper samples and as a result marginal hardness increase was recorded.

Ni and Alpas [32] have shown the formation of sub-micron size crystallites in the chips produced by dry machining of copper. Initial Vickers hardness of the workpiece in this case was 66.8 and the average hardness of the chips went to a value of 101.3. Interestingly, the machining chips that correspond to the hardness of 101.3 have shown occurrence of dynamic recovery and also dynamic recrystallization. It is noteworthy that the hardness of the dynamically recovered and recrystallized chips did not drop below the initial hardness of the workpiece. Our results from wear studies at LN_2 and also from rolling experiments at ambient temperature show similar trend. Various severely deformed materials are known to undergo such microstructural reconstitution because of the restoration processes (dynamic recovery and recrystallization), depending on the stacking fault energy, level of strain, strain rate, and the thermal activation available during concurrent deformation. In fact, Hughes and Hansen [9] were able to produce ultra-fine grains using a friction technique. Layers of fine grain (150–200 nm) have also been revealed in machined sub-surface of tantalum workpiece using focused ion beam [33]. Characterization of the microstructure of the sub-surface layer is in progress using a focused ion beam imaging technique. This technique has the capability of stress-free machining of a thin layer of material at the surface using ions and imaging microstructures with a resolution of the order of 5 nm.

Conclusions

A number of ball-on-disc sliding wear tests were conducted on work-hardened high purity copper samples against bearing steel in LN_2 environment at varying load (10, 15, and 20 N) and sliding speed (0.89, 1.11, and 1.34 m/s) conditions. The following conclusions can be drawn from the present study:

(a) Within the chosen window of test conditions, the steady state μ_F varied over a wide range (0.1 and 0.5). Relatively stable μ_F was recorded at the highest load of 20 N, irrespective of the sliding speed, and μ_F values were always less than 0.2. No clear correlation between μ_F and the sliding speed/load was found within the given test conditions.

(b) The measured wear rate was in the order of 10^{-4} mm^3/Nm and showed a decreasing trend with increasing load for intermediate (1.11 m/s) and highest (1.34 m/s) sliding speeds. A maximum wear depth of $\sim$30 μm was measured in the severely worn region after testing at a sliding speed of 1.34 m/s under 20 N load.

(c) SEM analysis of the worn samples showed that the copper surface was worn because of the plowing-induced severe mechanical damage. At lower load/sliding speed conditions, copper surface was severely damaged by abrasion or plowing and delamination of the tribo-layers. In contrast, at higher load and higher speed conditions, discontinuous delamination with large cavities, indicating severe deformation, was observed in the entire wear track.

(d) Despite severe test conditions, no substantial evidence of oxidative wear or the formation of mechanically mixed tribo-layer at the tribological interface was found. The characteristics of the sliding-induced severe deformation and damage accumulation had been discussed in the light of high contact stresses (more than 500 MPa) and thermal conditions (subzero flash temperature) at the tribological interface.

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